INTERLABORATORY STUDY 90-5

VOLATILE ORGANICS
(MISA TEST GROUPS 17 & 18)
IN REAGENT WATER

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VOLATILE ORGANICS (MISA TEST GROUPS 17 & 18) IN REAGENT WATER

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1 SUMMARY OF INTERLABORATORY STUDY 90-5

Interlaboratory Study 90-5 was initiated as part of an on-going program of laboratory performance management studies conducted by the Quality Management Office of Laboratory Services Branch, Environment Ontario. It assesses the performance of participating laboratories for the analysis of selected organic parameters (Table I, Section 3.1). Thirty-one laboratories (government, academic, industry, and commercial) agreed to participate in this study. Results were received from twenty-eight participants. The parameters were chosen from two different MISA Analytical Test Groups[1] - Groups 17 and 18.

The results from this study indicate that most laboratories are able to achieve acceptable within-laboratory precision or repeatability. This has improved for the parameters in this study compared to a previous study conducted one and a half years previous[4]. The major difference among the participating laboratories is slope bias, usually attributable to a difference in standards. This stresses the need for analytical laboratories to compare their in-house standards to reference standards so that comparability improves among laboratories.

2 INTRODUCTION

Interlaboratory performance studies are conducted to assess the comparability of data among different laboratories. These studies are useful for the identification of biases, precision and accuracy problems. Participation in such studies can serve as a guide for improving individual laboratory performance and maintaining performance standards. The Quality Management Office, Laboratory Services Branch, Environment Ontario has instituted an on-going program of interlaboratory studies to assess and enhance the performance of environmental laboratories providing analytical services.

This study was designed to assess the analytical variability among laboratories for the analysis of Non-halogenated and Water Soluble Volatile Organics. Parameters were chosen from analytical test groups listed in the MISA (Municipal and Industrial Strategy for Abatement) General Regulation[1]. Participants were requested to use methods which conformed to the MISA analytical principles and protocols given in the General Regulation[1].

Thirty-one laboratories (government, academic, industry, and commercial) agreed to participate in this study. Results were received from twenty-eight participants. A list of participants is given in Appendix 2. Each participant was assigned a unique identification code to maintain confidentiality.

A set of four samples was distributed to each of the thirty-one participants. The samples consisted of a reagent water blank and three reagent water samples fortified with subsets from MISA Test Groups 17 and 18[1]. Section 3 describes sample preparation, sample distribution, analytical methodology, and data evaluation procedures. Final results are tabled in Appendix 1 and discussed in Section 4.

3 PROCEDURE

3.1 Preparation of Samples

A stock spiking solution consisting of parameters from MISA Test Groups 17 and 18 was prepared in methanol. Table I lists the parameters and MISA Test Group.

TABLE I

PARAMETER	MISA TEST GROUP
Benzene	17
Toluene	17
o-Xylene	17
m-Xylene	17
p-Xylene	17
Acrylonitrile	18

Deionized, distilled water was used for the sample matrix. Care was taken that no plastic materials came into contact with the water when transferring to 250 mL glass bottles. Each bottle was filled to the top to eliminate headspace. Unspiked bottles were labelled "VOC1" and capped immediately.

All sample spiking was done with the appropriate size of microlitre syringe using a repeating dispenser. The low spike was labelled "VOC2", the mid-range spike was labelled "VOC3", and the high spike was labelled "VOC4". All samples bottles were individually spiked with the combined stock solution and immediately capped, allowing no headspace. Target values for each parameter are given in Appendix 1.

3.2 Sample Distribution

Samples were packed and stored overnight at 4°C. Samples were shipped the following day by Purolator Courier to the participating laboratories. A list of the laboratories receiving sample sets is given in Appendix 2. Samples were shipped on June 5, 1990. A copy of all correspondence is also included in Appendix 2.

3.3 Analytical Methodology

Participating laboratories were requested to analyze the samples using routine in-house methods that complied with the principles and protocols outlined in Schedule 3 of the MISA General Regulation[1]. Participants were requested on the report form provided (Appendix 2) to summarize their Sample Preparation Principles, Instrumental Measurement Method Principles, and indicate the Gas Chromatographic Column used. All participants were assigned a unique identification code.

3.4 Data Evaluation Technique

Results were submitted to the Quality Management Office, LSB in written form. All data were manually entered by laboratory code into an electronic spreadsheet. Blank spaces were left when a laboratory did not report results for a specific parameter that was present in the spiking material. Not all of the participants reported results for Acrylonitrile.

Laboratory 1010 had a different elution pattern for the three Xylenes from that reported by all of the other participants. Half of the combined result for o- & m-Xylenes was assigned to o-Xylene and half of this combined result was added to the reported result for p-Xylene.

The participating laboratories who reported results were mailed a copy of the tables of results on September 6, 1990. Two participants reported corrections to their data. Both sets of results are included in the tables, though the corrected results are used in the evaluation. Laboratory 1026 requested that their results be withdrawn as they had discovered a problem with their calibration standards. The results are retained in Tables 1-5 but are not included in any of the evaluation procedures outlined below.

The mean, median and standard deviation were calculated for each parameter in each sample and are included in Table 11, Appendix 1. Results were converted to percent recovery of the target value and are given in Tables 6-10, Appendix 1.

The results were evaluated using the technique outlined in Reference 2. This technique is summarized as follows:

1. Two samples at different concentrations are split among a number of laboratories and analyzed as requested. Results are entered into an electronic spreadsheet and programs (macros) are coded to perform the manipulations. The evaluation technique is performed after the participants have verified their results (see above).

2. High sample data evaluation:
   - i) Reject all results which differ from the median (H_m) by more than 10%.
   - ii) Calculate median (H), mean and standard deviation (S_h).
   - iii) Reinclude results if within 3 times S_h.
   - iv) Reiterate ii) and iii) until no further results are included.
   - v) Calculate relative standard deviation of final selected results (CV_h).

3. Low sample data evaluation:
   - i) Use 3 X CV_h X median(L_m) to exclude possible outliers.
   - ii) Calculate median (L), mean and standard deviation (S_l).

iii) Reinclude results if within 3 times S_l.
iv) Reiterate ii) and iii) until no further results are included.

4. Paired sample performance criteria:
   i) Examine ratio of S_h/S_l: If <2, use results as reported in concentration units. Otherwise convert results to percent recovery based on target value, if known, or use recovery relative to median value - H, L.
   ii) Prepare paired sample scatter diagrams of all results (Youden plots[3]).
   iii) Calculate perpendicular distances from each point to the two 45° lines (Slope and Intercept error lines) and select the lesser of the two perpendicular distances (PD).
   iv) Determine the median PD.
   v) Determine the mean of all PD values less than 2.5 times the median and use this mean to estimate the repeatability S_w.
   vi) Set warning limits for repeatability: 2 times S_w.
   vii) Set control limits for repeatability: 3 times S_w.
   viii) Set warning limits for possible bias: 3 times S_w (same as vii).
   ix) Set control limits for possible bias: 4.5 times S_w.

5. Code performance and summarize in table
   i) In upper left or lower right quadrant - Erratic
   ii) In lower left or upper right quadrant - Biased Low or High
   iii) On horizontal or vertical axis - Out of Control
   iv) On diagonal line through origin - Slope or Standard problem
   v) On diagonal line not through origin - Intercept or blank problems.

In this study, three spiked samples were provided to each participant. Samples VOC2 and VOC3 were designed at similar concentrations. The results are interpreted in pairs as follows: VOC2 (low) vs. VOC4 (high) and VOC3 (low) vs. VOC4 (high). The results for the spiked samples were not corrected for any background levels detected in the unspiked sample (VOC1), as only two positive values were reported. The statistical summary produced by this evaluation technique is given in Table 11.

The performance (Step 5) for each laboratory is summarized in Table 12. Note that laboratories flagged with slope (5. iv) or intercept (5. v) error are also rated according to their degree of precision. Laboratories within one S_w of the Slope or Intercept line are flagged with a capital "S" or "I". Laboratories between 1 and 2 S_w are flagged with a small "s" or "i". Laboratories that are further than 2 S_w from either line are just flagged H or L (See Reference 2 for further details).

The paired results have been plotted using the Youden technique[3]. Each plot includes the diagonal lines calculated in Step 5 iv) and v). The plots

also include the warning and control limits for repeatability and possible bias, centred on the target value. The inner circle represents the warning limits for repeatability (4 vi). The outer circle represents the control limits for repeatability (4 vii) and the warning limits for possible bias (4 viii). The outer arcs in the upper right and lower left quadrants represent the control limits for possible bias (4 ix). These diagrams are included in Appendix 1.

4 DISCUSSION

Results were received from twenty-eight of the thirty-one laboratories receiving samples. After the table of results was submitted to the participants for verification, Laboratory 1026 withdrew their results. They were not included in the evaluation procedure.

OVERVIEW OF INTERLABORATORY PERFORMANCE

The results from this study demonstrated that most of the participating laboratories are capable of maintaining good within laboratory precision or repeatability.

Very few of the participants were flagged as having erratic results for all parameters in this study, though some participants had problems with one or two parameters (Table 12). The analysis for Acrylonitrile appears to be the most difficult, having the greatest variability in the results. The points fall along a line which does not pass through the target values. This may be caused by calibration curvature. In this case, curvature may be induced by its high level of water solubility. This would also affect recovery, making it difficult to purge. Over recovery may be due to interferants co-eluting with Acrylonitrile.

The slightly greater variability demonstrated by m- & p-Xylenes may be due to the problem of co-elution of the two isomers. The different Gas Chromatographic columns used by the participants will have varying degrees of resolution of the isomers, which will result in variable quantitation.

Four of the five parameters in this study were included in Interlaboratory Study 89-1, conducted in January-February 1989[4]. Acrylonitrile was not included in the earlier study. The overall between-laboratory variability has improved in this study as compared to the previous study. The spiking levels used in this study were similar to the previous study. The standard deviation is smaller for the comparable sample-parameter combinations in this study, except for o-Xylene. There is no clear explanation why performance has not improved for this parameter.

The major source of variation among the participating laboratories appears to be related to slope bias, usually attributable to a difference in standards. This is also easily seen visually in Figures 1-10 as many data points are close to the diagonal line passing through the origin. Closeness of fit to the line reduces the liklihood that this bias is attributable to variable recovery. As with the previous study[4], this

source of variability stresses the need for analytical laboratories to validate their in-house standards with reference standards.

Very few individual parameters for a few of the laboratories were flagged as having possible intercept error.

INDIVIDUAL LABORATORY PERFORMANCE

Table 12 in Appendix 1 summarizes the individual performance for each paired evaluation, on an individual parameter and laboratory basis. The elution order of parameters is ranked as follows: Acrylonitrile, Benzene, Toluene, m- & p-Xylenes, and o-Xylene. A review of each participating laboratory follows.

Laboratory 1001

This laboratory's performance appears affected by the elution order of the parameters. The early eluting parameters (Acrylonitrile and Benzene) are flagged high while the later eluting Xylenes are biased low. Review of the Gas Chromatographic temperature program may solve this problem.

Laboratory 1002

The performance of this laboratory is variable. No pattern is evident based on elution order. The flagged results indicate a possible slope problem. Comparison of in-house standards with reference standards is advisable.

Laboratory 1003

This laboratory is flagged as biased high with a possible slope problem. Comparison of in-house standards with reference standards is advisable.

Laboratory 1005

The performance of Laboratory 1005 is variable. No pattern is evident based on elution order. The flagged results indicate a possible intercept problem for Toluene (a common laboratory solvent), though other parameters are erratic. Comparison of in-house standards with reference standards is advisable.

Laboratory 1006

This laboratory's performance was variable, with a tendency to being biased low. No pattern related to elution order was evident. Comparison of their in-house standard with reference standards may help improve their performance.

Laboratory 1007

This laboratory is biased low for all parameters. Some erratic performance was observed for Benzene and m- & p-Xylenes. Comparison of in-house standards with reference standards is advisable.

Laboratory 1009

The performance of Laboratory 1009 appears affected elution order. The early eluting parameters are biased low while the performance for the three Xylenes is acceptable. Adjustment of the Gas Chromatographic temperature program may help improve the recovery of the more volatile compounds.

Laboratory 1010

The performance of this laboratory was acceptable for three parameters but they had problems with the three Xylenes. This laboratory used a packed column, unlike all but two other participants, who used various capillary columns. Their packed column produced a different elution order for the Xylenes as compared to the other participants (see Section 4.4), which may account for a possible source of variability.

Laboratory 1013

This laboratory was biased low for several of the results, except for Acrylonitrile. Comparison of in-house standards with reference standards is advisable and may help improve performance.

Laboratory 1014

This laboratory's performance appears affected by the elution order of the parameters. The early eluting parameters (Acrylonitrile and Benzene) are flagged high while the later eluting Xylenes are biased low. Review of the Gas Chromatographic temperature program may solve this problem.

Laboratory 1015

This laboratory did not report results for sample VOC2, nor did they analyze for Acrylonitrile. They had acceptable performance for Toluene. Their results for the other compounds were biased low. Comparison of their in-house standard with reference standards may help improve their performance.

Laboratory 1016

This laboratory did not analyze for Acrylonitrile. Their performance was flagged as out of control or erratic because of the low results for sample VOC4, the high spike. If this sample was outside the normal calibration range and subsequently diluted, this may have introduced greater variability into the analysis, resulting with the flagged parameters. Comparison of their in-house standard with reference standards may help improve their performance.

Laboratory 1017

This laboratory's performance was acceptable, though there is a tendency to being biased high. No pattern related to elution order was evident. Comparison of their in-house standard with reference standards may help improve their performance for the Xylenes.

Laboratory 1018

This laboratory did not detect Acrylonitrile in the samples at the level with which they were spiked (below their laboratory detection limit). Their performance was acceptable for Toluene and o-Xylene but was biased low for the other two parameters. This laboratory used headspace analysis as opposed to Purge-and-trap analysis used by the other participants. The spiking level used for this study are at the lower end of detection for this analytical technique, which may account for the variability of performance for the two flagged parameters (Benzene and m- & p-Xylenes).

Laboratory 1019

The performance for this laboratory was biased low for all parameters except Acrylonitrile, which was biased high. If the same method was used for all parameters, it does not appear to perform in a similar manner for Acrylonitrile as compared to the other four parameters. This should be investigated. Comparison of their in-house standard with reference standards may also help improve their performance.

Laboratory 1020A and 1020B

Laboratory 1020 analyzed the samples using two different techniques. Acrylonitrile was not analyzed for by either technique. The "A" set were analyzed using HALL and PID detectors. The performance using this technique was biased high. The second technique ("B" set) used an MSD detector. The results from this technique were variable, with acceptable performance for Benzene but high and low flags for the other three parameters. If the same calibration standard(s) was used for both techniques, then the two techniques have not been designed to produce equivalent results and this should be investigated.

Laboratory 1021

This laboratory's performance appears affected by the elution order of the parameters. The early eluting parameters (Acrylonitrile, Benzene and Toluene) are acceptable while the later eluting Xylenes are biased high. Review of the Gas Chromatographic temperature program may solve this problem.

Laboratory 1022

This laboratory did not analyze for Acrylonitrile. This laboratory used a different technique for analysis that does not conform the Principles and Protocols outlined in the MISA General Regulation[1]. They used liquid-liquid extraction with CS_2 followed by analysis using GC/FID (MISA requires the purge-and-trap technique for sample preparation. GC/FID is an acceptable instrumental technique.) This laboratory's performance is flagged as biased low. There are several possible sources for this bias. There may be a difference in standards. These should be compared to reference standards. During the extraction procedure there may be losses of the target compounds during the concentration step. Use of an internal standard will help monitor extraction efficiency and/or losses and possibly correct a source of low bias.

Laboratory 1023

This laboratory's performance was biased high except for m- & p-Xylenes. As they used a packed column as opposed to a capillary column (as used by most of the other participants), there may be a difference in the elution order of the Xylenes that was not accounted for. This should be investigated. Comparison of their in-house standard with reference standards may also help improve their performance.

Laboratory 1024

Laboratory 1024 does not analyze for Acrylonitrile. Their performance was biased low. Comparison of their in-house standard with reference standards may help improve their performance. This laboratory used headspace analysis (not a MISA protocol[1]), which may also account for their low bias as this technique is not as efficient at low levels.

Laboratory 1025

Laboratory 1025 did not have sufficient calibration standard available to analyze these samples for Acrylonitrile, though they routinely perform this test. Their performance was erratic, with some parameters having acceptable results for some samples, but being out of control for different sample-parameter combinations. Consistency in applying their analytical procedure should help improve their performance.

Laboratory 1026

Withdrew their results. No evaluation of performance.

Laboratory 1027

This laboratory's performance was biased low. They did not analyze for Acrylonitrile. Comparison of their in-house standard with reference standards is advisable and may help improve performance.

Laboratory 1028

This laboratory had acceptable performance for one pair comparison for Benzene but was biased low for the other parameters. They did not analyze for Acrylonitrile. Comparison of their in-house standard with reference standards may help improve their performance.

Laboratory 1029

This laboratory did not analyze for Acrylonitrile. Their performance was erratic, with some parameters having acceptable results for Toluene, but being out of control or biased low for different sample-parameter combinations. Consistency in applying their analytical procedure should help improve their performance. Comparison of their in-house standard with reference standards may also help improve their performance.

Laboratory 1030

This laboratory did not analyze for Acrylonitrile. Their performance was erratic, with some parameters having acceptable results for Benzene, but being out of control or biased low for different sample-parameter combinations. Consistency in applying their analytical procedure should help improve their performance. Comparison of their in-house standard with reference standards may also help improve their performance.

Laboratory 1031

This laboratory's performance was biased low. They did not detect o-Xylene in any of the spiked samples. They did not analyze for Acrylonitrile at the time this study took place. Comparison of their in-house standard with reference standards may help improve their performance. The lack of identification of o-Xylene at levels well above their MDL (MDL = 0.5 ppb) is also a matter of concern.

Laboratory 1032

This laboratory did not analyze for Acrylonitrile. Their performance was erratic. Consistency in applying their analytical procedure should help improve their performance.

5 REFERENCES

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6 APPENDIX 1

Tables 1-5	Sample Results (µg/L)
Tables 6-10	Sample Results as Percent Recovery of Target Value
Table 11	Statistical Summary
Table 12	Summary of Individual Laboratory Performance
Figures 1-10	Youden Plots

TABLE 1: ACRYLONITRILE (Results in µg/L)

CODE	VOC1	VOC2	VOC3	VOC4
TARGET		5.8	7.8	16.6
1001	<0.5	15	16	35
1002	<1.90	5.83	7.93	18.11
1003	<2.0	7.2	10.4	22.6
1005	ND	4.99	6.79	17.55
1006	<DL	9.8	16.2	30.5
1007	<DL	1.4	2	4.9
1009	<MDL	4.86	6.60	13.80
1010	<.7	4.0	6.2	15.1
1013	ND	6.4	8.4	21
1014	<5	7.4	20	46
1015	N/A	N/A	N/A	N/A
1016	N/A	N/A	N/A	N/A
1017	ND	5.6	7.7	20
1018	<100	<100	<100	<100
1019	<1.0	20.2	22.0	101.6
1020A	N/A	N/A	N/A	N/A
1020B	N/A	N/A	N/A	N/A
1021	<0.3	5.5	8.0	16
1022	N/A	N/A	N/A	N/A
1023	<1.0	20	24	49
1024	N/A	N/A	N/A	N/A
1025		N/A	N/A	N/A
1026**	0.98	15.6	23.0	46.0
1027	N/A	N/A	N/A	N/A
1028	N/A	N/A	N/A	N/A
1029	<DL	<DL	<DL	7.1
1030	N/A	N/A	N/A	N/A
1031	N/A	N/A	N/A	N/A
1032	N/A	N/A	N/A	N/A

** Laboratory 1026 withdrew their results from this study.

TABLE 2: BENZENE (Results in µg/L)

CODE	VOC1	VOC2	VOC3	VOC4
TARGET		5.9	7.8	16.6
1001	<0.2	10	11	23
1002	<.24	6.35	8.32	17.03
1003	<0.1	6.7	9.5	21.0
1005	ND	5.81	6.65	16.0
1006	<DL	5.6	7.0	15.4
1007	<DL	0.5	6.6	12.0
1007*		5.1		
1009	<MDL	5.60	7.42	15.74
1010	<.5	6.1	7.9	16.8
1013	ND	5.8	7.4	16.2
1014	<.37	7.5	9.8	20
1015	<MDL	NA	4.7	11
1016	<0.5	5.8	7.7	14.1
1017	ND	7.4	8.0	18
1018	<2	5	7	14
1019	<0.5	4.4	5.4	12.2
1020A		6.6	8.6	18.5
1020B		5.8	8.0	17.0
1021	<0.1	5.9	8.4	18
1022	<0.07	2.17	3.33	3.93
1023	<0.5	6.7	10	23
1024	0.35	3.16	3.71	8.71
1025	<0.5	5.58	7.00	17.4
1026**	0.14	26.2	35.2	79.1
1027	ND	4.73	6.26	13.57
1028	<1.0	5.5	7.3	15.9
1029	<DL	6.0	7.5	14.8
1030	ND	5.98	8.80	17.09
1031	<0.5	5.2	7.6	14.3
1032	ND	5.5	7.4	13.9

* Revised result for sample VOC2 (used for subsequent calculations).
** Laboratory 1026 withdrew their results from this study.

TABLE 3: TOLUENE (Results in µg/L)

CODE	VOC1	VOC2	VOC3	VOC4
TARGET		6	8	16.9
1001	<0.2	11	12	18
1002	<.39	4.45	6.45	14.53
1003	<0.2	7.7	10.4	21.3
1005	ND	6.25	6.93	16.22
1006	<DL	5.6	7.4	16.6
1007	<DL	4.5	6.1	11.8
1009	0.14	4.08	5.50	11.42
1010	<.2	6.3	8.2	17.3
1013	ND	5.6	7.2	15.6
1014	<0.29	5.6	8.3	16
1015	<MDL	NA	7.7	17
1016	<0.5	5.8	7.3	13.3
1017	ND	7.3	8.0	17
1018	<2	6	8	18
1019	<0.5	2.4	3.4	8.0
1020A		7.5	9.8	20.8
1020B		6.5	9.1	19.0
1021	<0.2	5.9	8.1	18
1022	<0.04	5.01	9.12	11.82
1023	<0.6	6.5	9.2	21
1024		4.03	5.00	11.47
1025	<0.5	5.99	7.48	18.3
1026**	0.75	25.9	36.2	82.1
1027	ND	5.25	6.97	15.86
1028	<1.0	5.1	6.8	15.0
1029	<DL	6.7	8.7	18.2
1030	ND	3.44	5.07	10.25
1031	<0.5	3.6	6.0	12.1
1032	ND	5.9	7.7	14.6

**. Laboratory 1026 withdrew their results from this study.

TABLE 4: m- AND p-XYLENES (Results in µg/L)

CODE	VOC1	VOC2	VOC3	VOC4
TARGET		8.7	11.6	33
1001	<0.2	6.9	7.2	14
1002	<0.48	9.75	12.76	27.45
1003	<0.2	9.7	12.6	28.0
1005	ND	9.66	11.14	23.28
1006	<DL	7.7	10.1	22.4
1007	<DL	6.7	8.5	14.8
1009	<MDL	8.63	11.96	24.93
1010	<.2	7.7	9.95	20.9
1013	ND	8.2	10.5	23
1014	<.68	7.5	10	21
1015	<MDL	NA	10	6.8
1016	<1.1	9.0	11.3	21.0
1017	ND	11	12	26
1018	<2	8	11	24
1019	<1.0	3.6	5.0	10.6
1020A		12.6	16.7	35.4
1020B		10.1	14.3	29.6
1021	<0.1	4.9	6.8	15
1021*	<0.1	9.8	13.6	30
1022	<0.01	3.88	5.79	6.99
1023	<0.7	3.7	6.5	12
1024		4.43	5.75	12.46
1025	<1.0	8.72	10.5	24.4
1026	0.26	42.3	58.2	126.7
1027	ND	8.22	10.66	22.65
1028	<1.0	6.5	9.0	18.7
1029	<DL	7.7	9.9	20.7
1030	ND	8.98	13.04	26.68
1031	<1.1	3.3	4.8	9.2
1032	ND	9.9	12.7	23.1

NOTE: Lab 1010 had a different elution pattern for the Xylenes. Half of the result reported for o- & p-Xylenes plus the result from m-Xylene was assigned for m- & p-Xylenes.

* Revised results for Laboratory 1021 (used for subsequent calculations).
** Laboratory 1026 withdrew their results from this study.

TABLE 5: o-XYLENE (Results in µg/L)

CODE	VOC1	VOC2	VOC3	VOC4
TARGET		3	4	8.5
1001	<0.2	2.5	2.8	5.7
1002	<0.42	3.58	4.84	10.89
1003	<0.2	3.6	4.7	9.8
1005	ND	3.48	4.30	7.92
1006	<DL	2.8	3.6	8.4
1007	<DL	1.6	2.1	4.6
1009	<MDL	3.00	4.18	8.86
1010	<0.2	4.3	5.55	11.7
1013	ND	3.1	4.0	8.6
1014	<0.39	2.4	3.5	7.3
1015	<MDL	NA	3.7	8.0
1016	<0.5	3.1	3.8	7.0
1017	ND	4.0	4.5	10
1018	<2	3	4	9
1019	<0.5	2.2	3.0	6.4
1020A		3.9	5.1	11.5
1020B		2.1	3.0	6.5
1021	<0.1	3.8	5.3	12
1022	<0.01	1.76	2.28	2.47
1023	<0.7	9.7	12	33
1024		2.42	3.02	6.79
1025	<0.5	3.33	3.87	9.00
1026**	0.055	15.9	22.0	47.8
1027	ND	2.93	3.80	8.15
1028	<1.0	1.3	1.8	4.3
1029	<DL	3.0	3.7	7.7
1030	ND	2.78	3.87	8.47
1031	<0.5	<0.5	<0.5	<0.5
1032	ND	3.6	4.7	9.1

** Laboratory 1026 withdrew their results from this study.

TABLE 6: ACRYLONITRILE AS PERCENT RECOVERY OF THE TARGET VALUE

CODE	VOC2	VOC3	VOC4
TARGET	6.1	8.1	17
1001	246%	198%	206%
1002	96%	98%	107%
1003	118%	128%	133%
1005	82%	84%	103%
1006	161%	200%	179%
1007	23%	25%	29%
1009	80%	81%	81%
1010	66%	77%	89%
1013	105%	104%	124%
1014	121%	247%	271%
1017	92%	95%	118%
1019	331%	272%	598%
1021	90%	99%	94%
1023	328%	296%	288%
1029	<DL	<DL	42%

TABLE 7: BENZENE AS PERCENT RECOVERY OF TARGET VALUE

CODE	VOC2	VOC3	VOC4
TARGET	6.1	8.1	17.1
1001	164%	136%	135%
1002	104%	103%	100%
1003	110%	117%	123%
1005	95%	82%	94%
1006	92%	86%	90%
1007	84%	81%	70%
1009	92%	92%	92%
1010	100%	98%	98%
1013	95%	91%	95%
1014	123%	121%	117%
1015	NA	58%	64%
1016	95%	95%	82%
1017	121%	99%	105%
1018	82%	86%	82%
1019	72%	67%	71%
1020A	108%	106%	108%
1020B	95%	99%	99%
1021	97%	104%	105%
1022	36%	41%	23%
1023	110%	123%	135%
1024	52%	46%	51%
1025	91%	86%	102%
1027	78%	77%	79%
1028	90%	90%	93%
1029	98%	93%	87%
1030	98%	109%	100%
1031	85%	94%	84%
1032	90%	91%	81%

TABLE 8: TOLUENE AS PERCENT RECOVERY OF TARGET VALUE

CODE	VOC2	VOC3	VOC4
TARGET	6.2	8.3	17.4
1001	177%	145%	103%
1002	72%	78%	84%
1003	124%	125%	122%
1005	101%	83%	93%
1006	90%	89%	95%
1007	73%	73%	68%
1009	66%	66%	66%
1010	102%	99%	99%
1013	90%	87%	90%
1014	90%	100%	92%
1015	NA	93%	98%
1016	94%	88%	76%
1017	118%	96%	98%
1018	97%	96%	103%
1019	39%	41%	46%
1020A	121%	118%	120%
1020B	105%	110%	109%
1021	95%	98%	103%
1022	81%	110%	68%
1023	105%	111%	121%
1024	65%	60%	66%
1025	97%	90%	105%
1027	85%	84%	91%
1028	82%	82%	86%
1029	108%	105%	105%
1030	55%	61%	59%
1031	58%	72%	70%
1032	95%	93%	84%

TABLE 9: m- & p-XYLENES AS PERCENT RECOVERY OF TARGET VALUE

CODE	VOC2	VOC3	VOC4
TARGET	9.1	12.1	25.5
1001	76%	60%	55%
1002	107%	105%	108%
1003	107%	104%	110%
1005	106%	92%	91%
1006	85%	83%	88%
1007	74%	70%	58%
1009	95%	99%	98%
1010	37%	36%	36%
1013	90%	87%	90%
1014	82%	83%	82%
1015	NA	83%	27%
1016	99%	93%	82%
1017	121%	99%	102%
1018	88%	91%	94%
1019	40%	41%	42%
1020A	138%	138%	139%
1020B	111%	118%	116%
1021	108%	112%	118%
1022	43%	48%	27%
1023	41%	54%	47%
1024	49%	48%	49%
1025	96%	87%	96%
1027	90%	88%	89%
1028	71%	74%	73%
1029	85%	82%	81%
1030	99%	108%	105%
1031	36%	40%	36%
1032	109%	105%	91%

TABLE 10: o-XYLENE AS PERCENT RECOVERY OF TARGET VALUE

CODE	VOC2	VOC3	VOC4
TARGET	3.1	4.2	8.8
1001	81%	67%	65%
1002	115%	115%	124%
1003	116%	112%	111%
1005	112%	102%	90%
1006	90%	86%	95%
1007	52%	50%	52%
1009	97%	100%	101%
1010	139%	132%	133%
1013	100%	95%	98%
1014	77%	83%	83%
1015	NA	88%	91%
1016	100%	90%	80%
1017	129%	107%	114%
1018	97%	95%	102%
1019	71%	71%	73%
1020A	126%	121%	131%
1020B	68%	71%	74%
1021	123%	126%	136%
1022	57%	54%	28%
1023	313%	286%	375%
1024	78%	72%	77%
1025	107%	92%	102%
1027	95%	90%	93%
1028	42%	43%	49%
1029	97%	88%	88%
1030	90%	92%	96%
1031	0%	0%	0%
1032	116%	112%	103%

TABLE 11: STATISTICAL SUMMARY

	ACRYLONITRILE			BENZENE			TOLUENE			m- & p-XYLENES			o-XYLENE		
	VOC2	VOC3	VOC4	VOC2	VOC3	VOC4	VOC2	VOC3	VOC4	VOC2	VOC3	VOC4	VOC2	VOC3	VOC4
TARGET	6.1	8.1	17.0	6.1	8.1	17.1	6.2	8.3	17.4	9.1	12.1	25.5	3.1	4.2	8.8
MEAN	7.88	10.81	27.88	5.57	7.44	15.66	5.5	7.57	15.66	7.41	10.06	20.30	2.97	3.77	8.68
MEDIAN	6.12	8.2	19.055	5.8	7.46	15.95	5.8	7.59	16.11	8.2	10.5	22.53	3.0	3.87	8.28
STD DEV	5.817	6.933	23.193	1.722	1.728	3.932	1.912	1.741	3.325	2.827	3.062	7.517	1.662	0.940	5.369
n	14	14	15	27	28	28	27	28	28	27	28	28	26	27	27
SELECTED MEDIAN	5.6	8.2	17.83	5.8	7.5	16.1	5.7	7.48	16.22	8.1	10.5	22.4	2.69	3.8	8.15
SELECTED STD DEV	2.024	6.523	2.838	0.911	1.723	3.311	1.945	1.773	3.380	2.838	3.075	7.634	1.067	0.940	2.280
n	11	14	8	24	27	26	26	27	27	26	27	27	24	25	25

	ACRYLONITRILE		BENZENE		TOLUENE		m- & p-XYLENES		o-XYLENE	
	2 vs 4	3 vs 4	2 vs 4	3 vs 4	2 vs 4	3 vs 4	2 vs 4	3 vs 4	2 vs 4	3 vs 4
S_h/S_l	1.401	0.435	3.634	1.916	1.737	1.906	2.690	2.483	2.137	2.526
S_w	1.217	1.010	1.683	0.480	0.425	0.341	2.015	2.066	3.775	2.611

TABLE 12: SUMMARY OF INDIVIDUAL LABORATORY PERFORMANCE

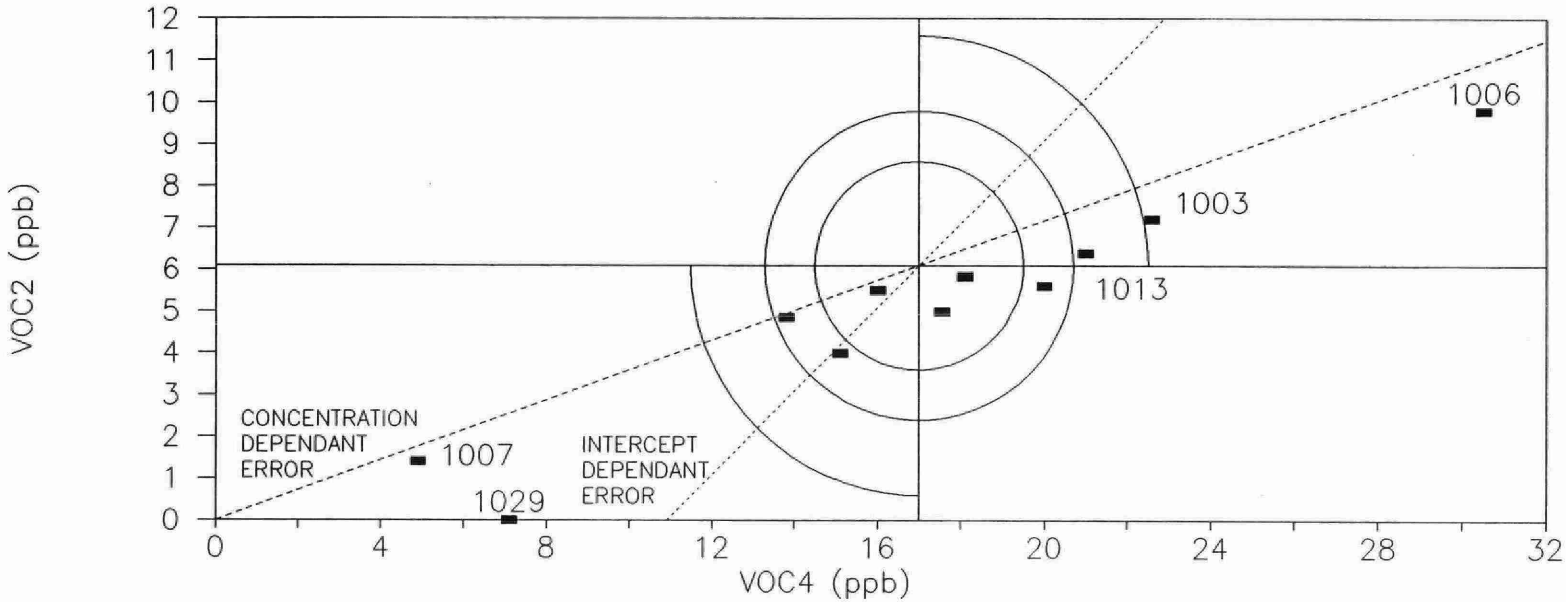
LAB	ACRYLONITRILE		BENZENE		TOLUENE		m- & p-XYLENES		o-XYLENE	
CODE	2 VS 4	3 VS 4	2 VS 4	3 VS 4	2 VS 4	3 VS 4	2 VS 4	3 VS 4	2 VS 4	3 VS 4
1001	Hs	HS	H	HS	OC	OC	Le	Ls	Le	LS
1002	A	A	Al	A	LS	Ls	HS	WHS	Hs	He
1003	HS	HS	He	HS	HS	HS	Hs	Hs	HS	HS
1005	A	A	LS	WLI	Al	LI	ER	Ls	ER	OC
1006	HS	Hs	LS	WLS	A	WLI	Ls	Ls	A	LI
1007	LS	LS	Le	Ls	LS	Ls	Le	Le	LS	LS
1009	A	WLS	LS	WLS	LS	LS	A	A	A	A
1010	A	A	A	A	A	A	LS	LS	Hs	HS
1013	WHS	WOC	WLS	A	WLS	LS	LS	Ls	Ls	A
1014	OC	Hs	H	HS	WLS	WOC	LS	LS	Ls	LS
1015				LS		A		Le		LS
1016			Le	OC	OC	Le	OC	Le	OC	Le
1017	Al	Al	Hi	A	Al	A	OC	A	HI	Hs
1018			LS	LS	A	A	LI	LI	A	A
1019	He	He	LS	LS	LS	Ls	LS	LS	LS	LS
1020A			HS	WHS	HS	HS	HS	HS	HS	He
1020B			A	A	WHS	HS	Hs	HS	Ls	LS
1021	A	A	OC	A	A	A	He	Hs	He	He
1022			Le	Le	Ls	ER	Le	Le	Le	Le
1023	Hs	HS	He	Hs	OC	He	L	Le	He	He
1024			LS	LS	LI	Ls	LS	LS	LS	LS
1025			OC	Al	WLS	ER	A	LI	A	OC

LAB	ACRYLONITRILE		BENZENE		TOLUENE		m- & p-XYLENES		o-XYLENE	
CODE	2 VS 4	3 VS 4	2 VS 4	3 VS 4	2 VS 4	3 VS 4	2 VS 4	3 VS 4	2 VS 4	3 VS 4
1027			LS	LS	WLS	LI	LS	LS	A	LS
1028			Ls	A	LS	LS	LS	LS	Ls	Ls
1029			OC	LS	A	A	Ls	LS	WOC	LS
1030			A	A	LS	LS	AI	WHI	A	WLI
1031			LS	OC	Ls	LS	LS	Ls		
1032			Le	OC	OC	OC	ER	ER	WHI	HI

KEY TO SUMMARY TABLE

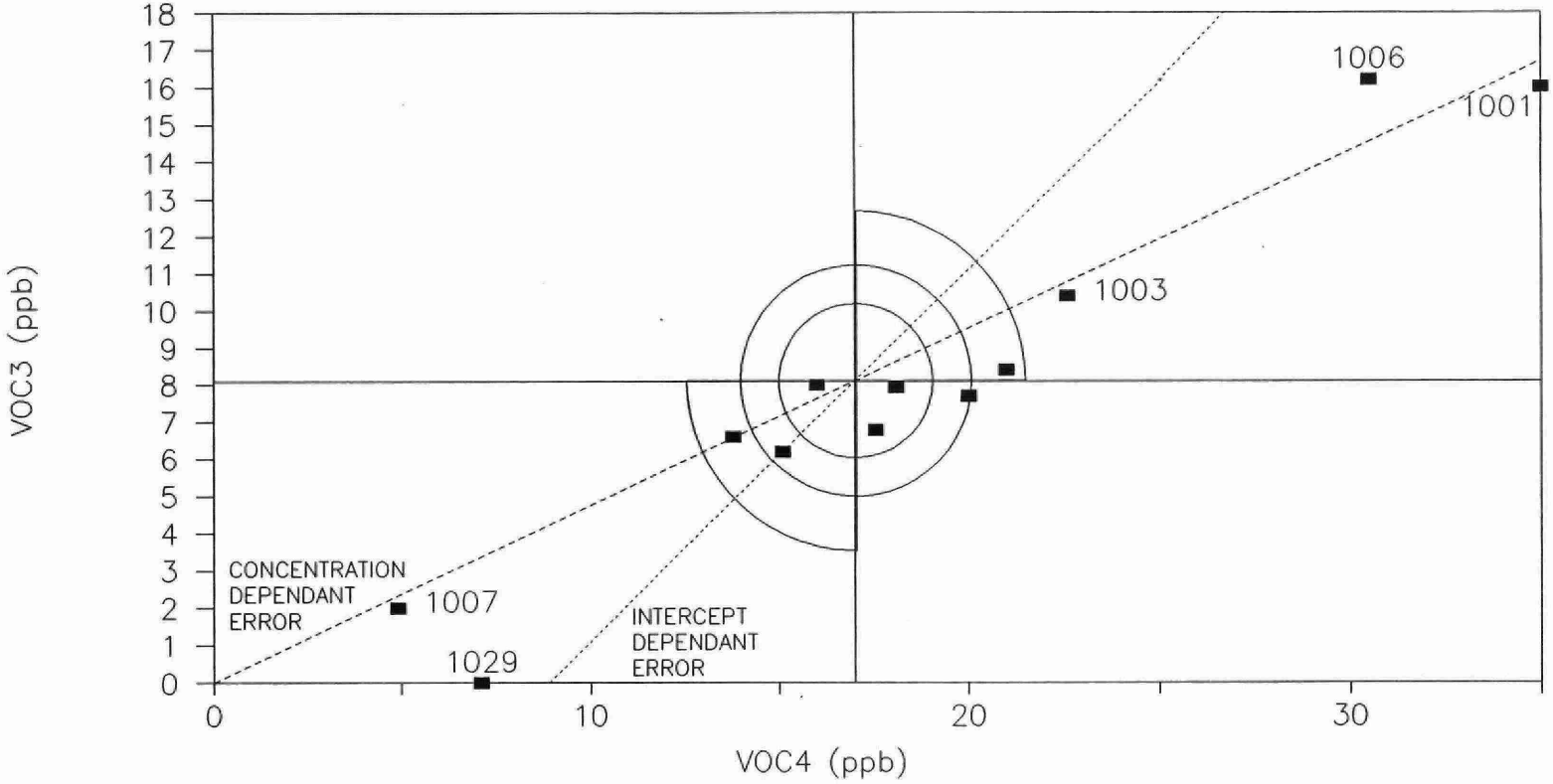
A	Acceptable Performance
WAI	Warning: Slight imprecision
WLI	Warning: Biased low, Probable intercept problem
WHI	Warning: Biased high, Probable intercept problem
WLS	Warning: Biased low, Probable slope problem
WHS	Warning: Biased high, Probable slope problem
WOC	Warning: Out of control - one result erratic
LI	Biased Low, Probable intercept problem
LS	Biased low, Probable slope problem
LI	Biased low, Possible intercept problem
Ls	Biased low, Possible slope problem
L	Biased low
Le	Biased low and/or erratic
HI	Biased high, Probable intercept problem
HS	Biased high, Probable slope problem
Hi	Biased high, Possible intercept problem
Hs	Biased high, Possible slope problem
H	Biased high
He	Biased high and/or erratic
OC	Out of control - one result erratic
ER	Both results erratic

FIGURE 1: ACRYLONITRILE – VOC2 vs VOC4



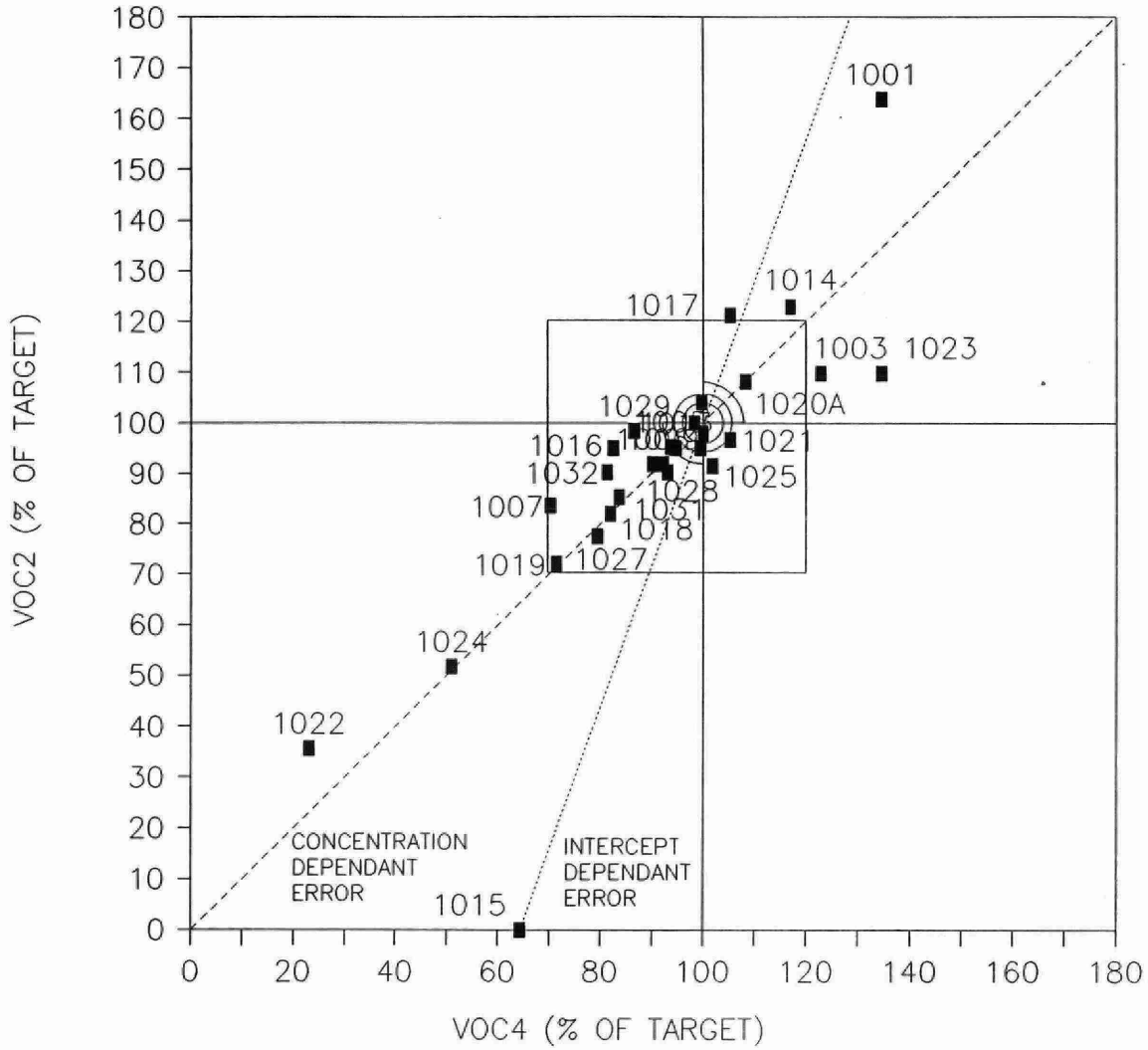
LABORATORIES 1001, 1014, 1019 & 1023 ARE OUTSIDE SCALE OF DIAGRAM.

FIGURE 2: ACRYLONITRILE – VOC3 vs VOC4



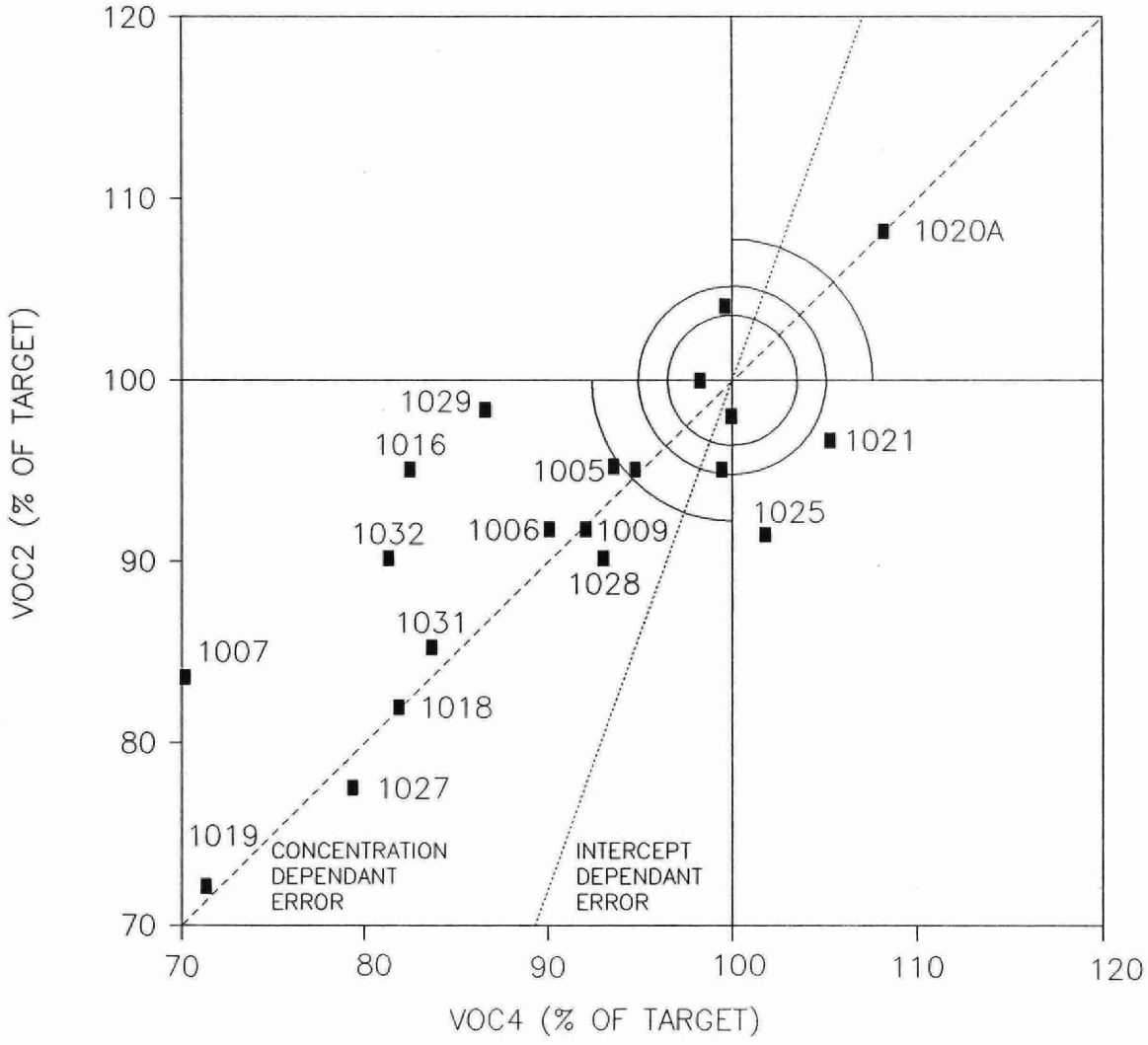
LABORATORIES 1014, 1019 AND 1023 ARE OUTSIDE SCALE OF DIAGRAM.

FIGURE 3: BENZENE – VOC2 vs VOC4



AREA INSIDE BOX IS EXPANDED IN FIGURE 3A

FIGURE 3A: BENZENE – VOC2 vs VOC4



EXPANDED BOX FROM FIGURE 3.

FIGURE 4: BENZENE – VOC3 vs VOC4

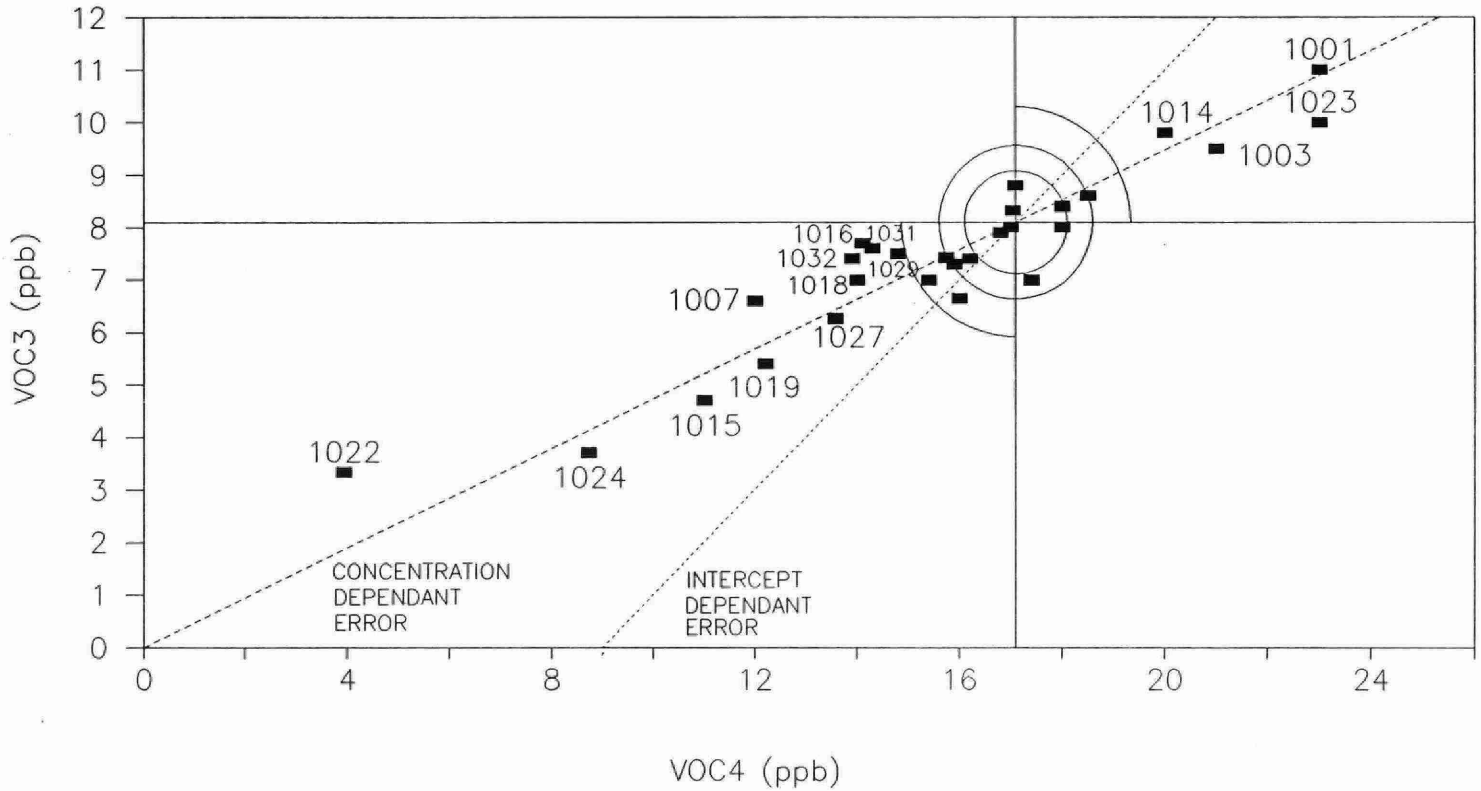


FIGURE 5: TOLUENE – VOC2 vs VOC4

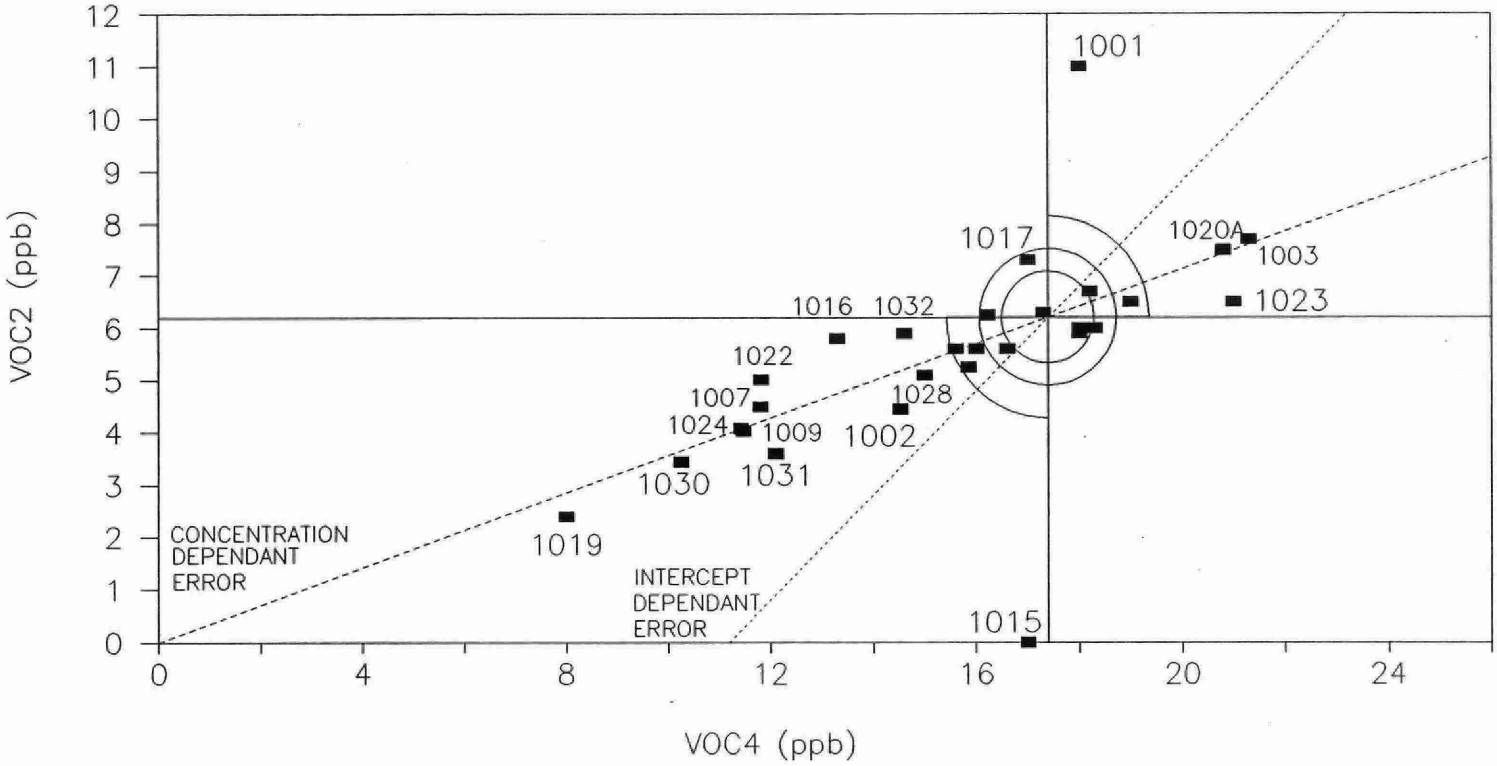


FIGURE 6: TOLUENE – VOC3 vs VOC4

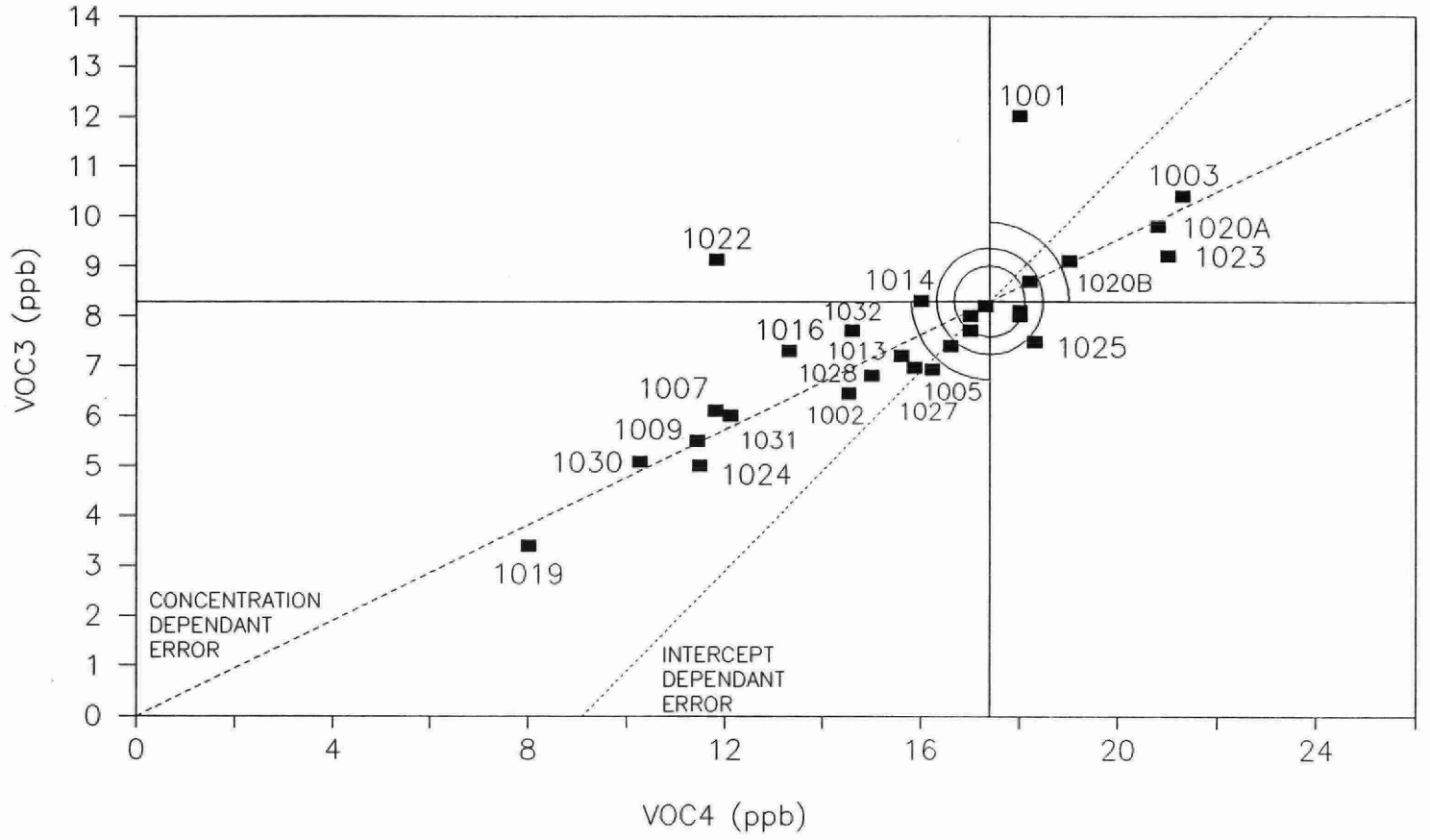


FIGURE 7: m- & p-XYLENES - VOC2 vs VOC4

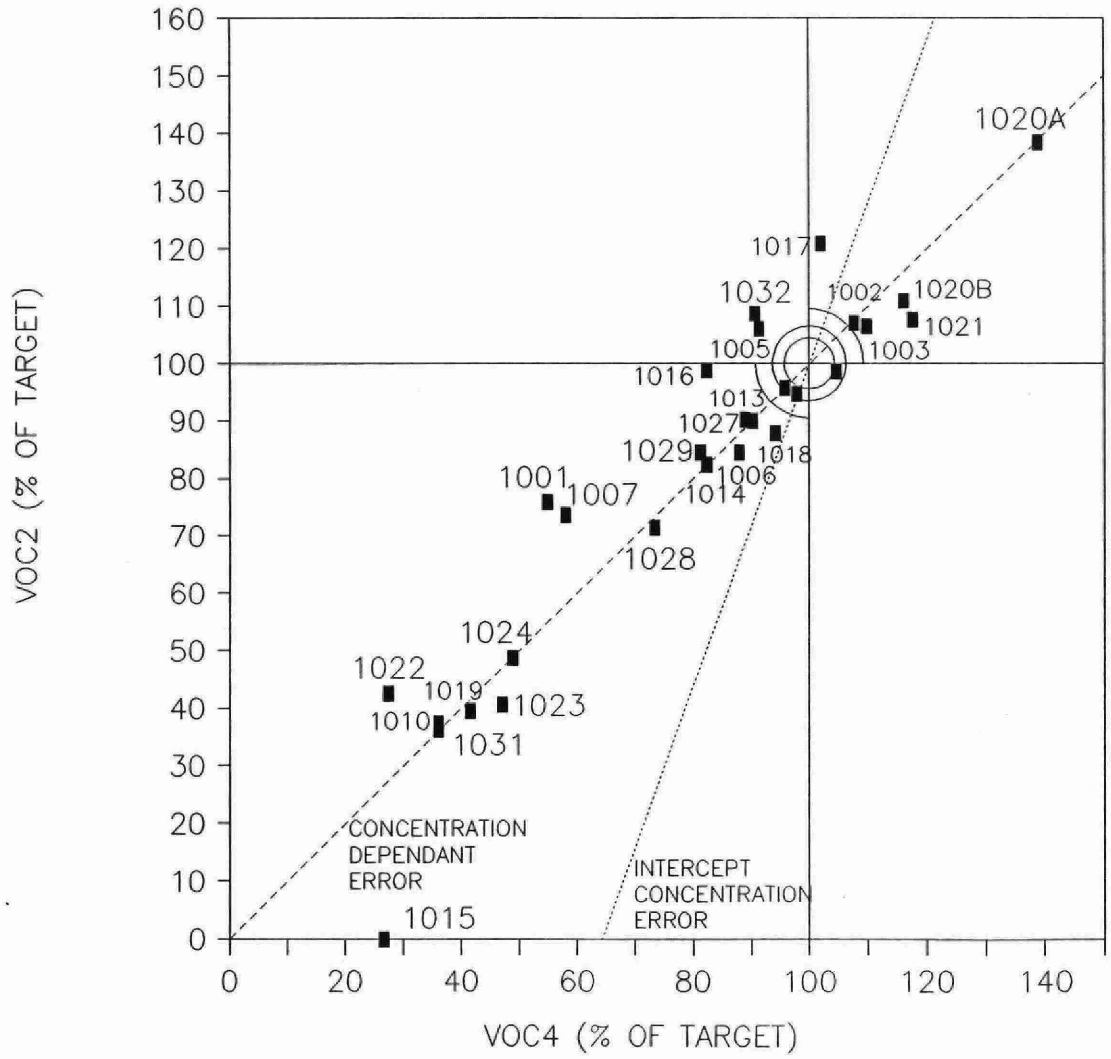


FIGURE 8: m- & p-XYLENES - VOC3 vs VOC4

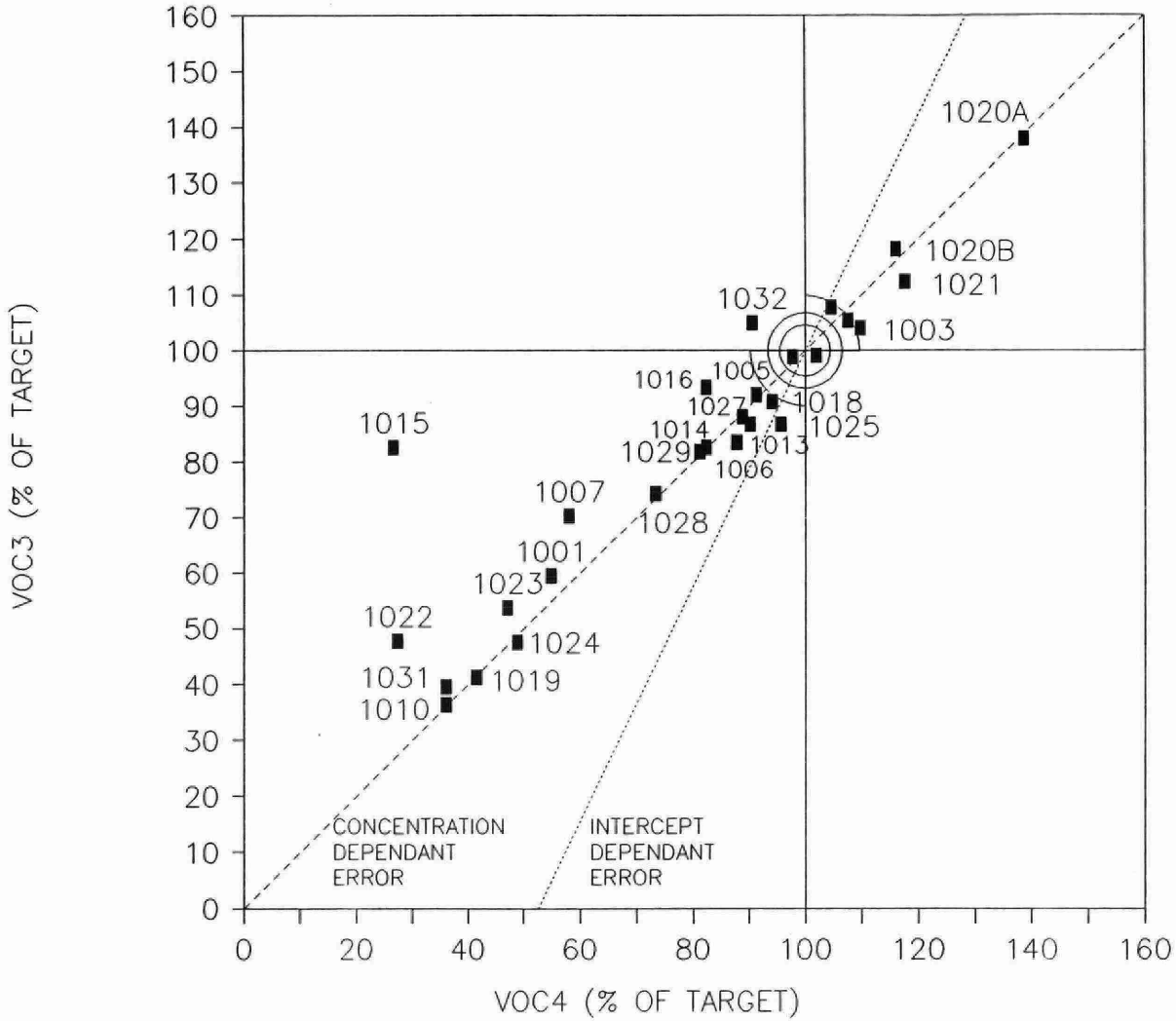
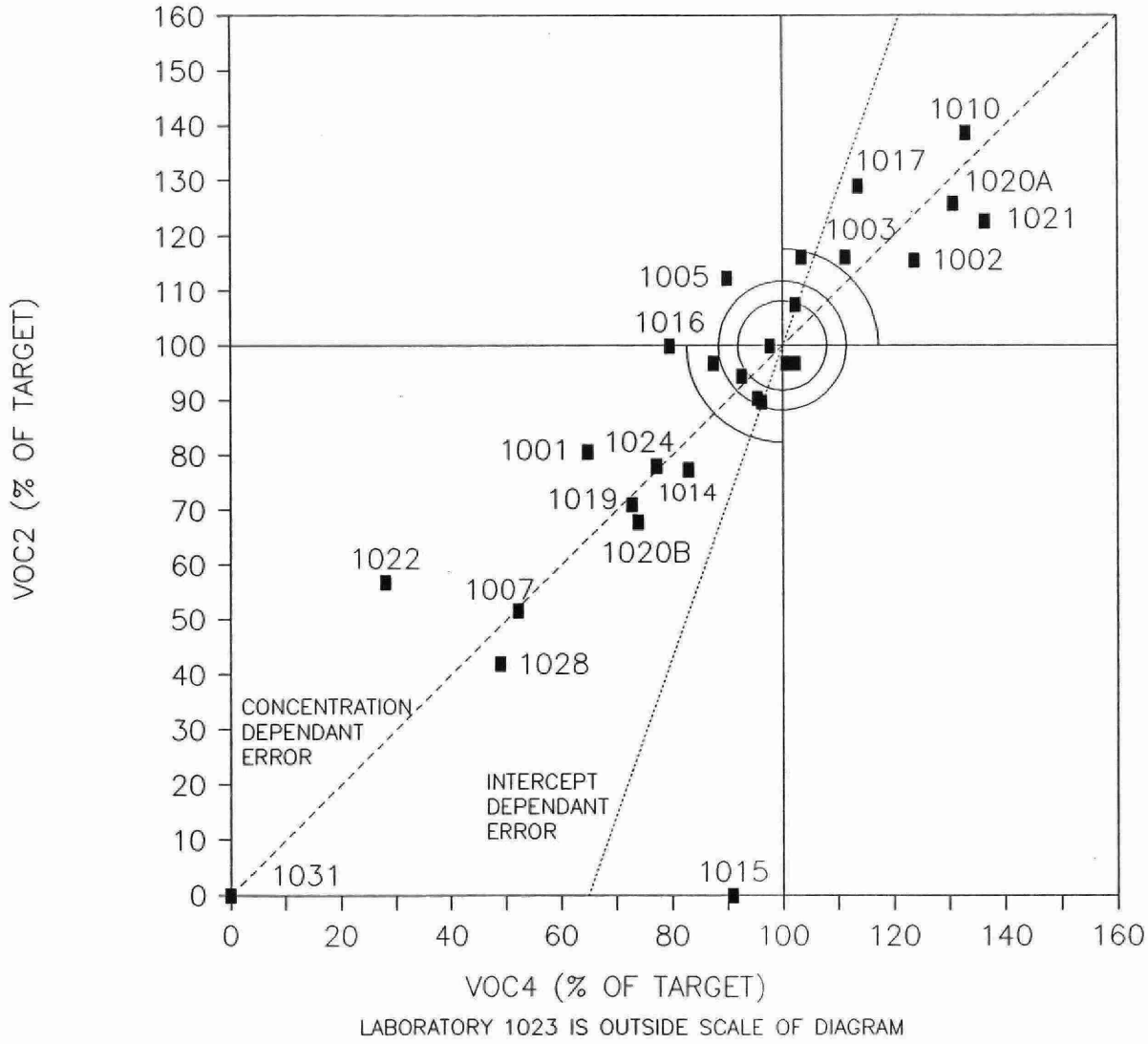
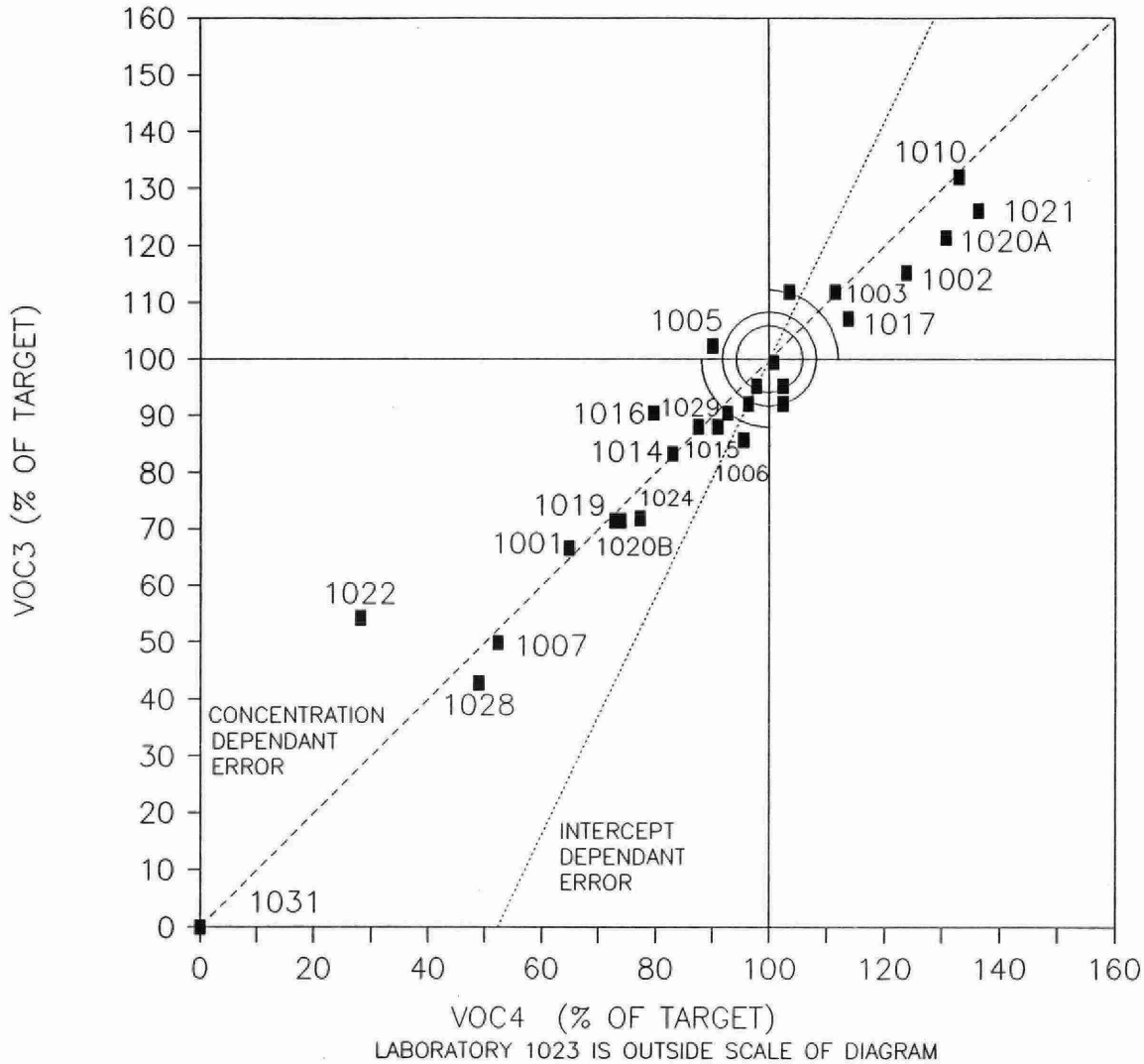


FIGURE 9: o-XYLENE - VOC2 vs VOC4



LABORATORY 1023 IS OUTSIDE SCALE OF DIAGRAM

FIGURE 10: o-XYLENE - VOC3 vs VOC4



7 APPENDIX 2

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MOE INTERLABORATORY VARIABILITY STUDY NOTIFICATION FOR THE ANALYSIS OF TRACE ORGANIC COMPOUNDS STUDY NO. 90-5

INTRODUCTION

Private laboratories receiving this notification are invited by Environment Ontario, Laboratory Services Branch to participate in an interlaboratory variability study of spiked reagent water, conducted using MISA analysis protocols. Laboratories interested in participating in this program, scheduled for the week of June 4, 1990, should return the attached response sheet by May 30, 1990 (FAX: (416) 235-5744). Any questions should be addressed to Sylvia Cussion at (416) 235-5842.

BACKGROUND

This study is being conducted to assist laboratories in assessing their analytical performance. All procedures should follow those principles and protocols outlined in the MISA regulations (Ontario Regulation 695/88 as amended to Ontario Regulation 533/89). Sample sets will include four samples: a blank, 2 samples in the range 5-10 times the MDL, and a high sample in the range 20-25 times the MDL.

NOTE: Any laboratory that does not have a copy of the MISA general regulations should contact Catherine Doehler at (416) 235-6055.

The following parameters are to be included in this interlaboratory study:

VOLATILES (to be analyzed by GC/MS and/or GC/ECD/FID)
MISA GROUPS 17 and 18

Benzene
Toluene
o-Xylene
m-Xylene
p-Xylene
Acrylonitrile

TIME LIMIT: 7 Days Storage

SCHEDULE

During the week of June 4, 1990, participating laboratories will receive a total of four samples for analysis. All samples will be spiked reagent water.

Participating laboratories are expected to analyze the samples within the time limits specified in Schedule 2 of the MISA General Regulations. Blank report forms will be provided with the samples. Results are to be reported within 30 days of receipt of samples to Sylvia Cussion at the following address:

Ministry of the Environment
Laboratory Services Branch
Quality Management Unit
P.O. Box 213, 125 Resources Rd.
Rexdale, Ontario
M9W 5L1

SUMMARY OF RESULTS

All participating laboratories will be assigned a unique identification code. All laboratories will receive a complete set of results where everyone will be identified only by their identification code. Recommendations made by the MOE will be provided in a final published report. Results will remain confidential and will only be released with the written permission of the individual participants.

It is the intent of this interlaboratory study (along with others) to assess the interlaboratory variability and detection capability for a broad range of organics and inorganics.

MOE INTERLABORATORY VARIABILITY STUDY NOTIFICATION FOR THE ANALYSIS OF TRACE ORGANIC COMPOUNDS

STUDY NO. 90-5

	YES	NO
We will participate in MOE Study 90-5 for the analysis of Volatiles (MISA Groups 17 and 18).	☐	☐

Minimum amount of sample volume required: _____

For the completeness of our records, and to avoid any shipping delays, please fill in the following:

Mailing Address:

_____

_____

_____

_____

_____

Shipping Address:

_____

_____

_____

_____

_____

Contact Person: _____

Phone Number: _____

Please return this response form before May 30, 1990 to:

Sylvia Cussion
Ontario Ministry of the Environment
Laboratory Services Branch
Quality Management Unit
P.O. Box 213, 125 Resources Rd.
Rexdale, Ontario
M9W 5L1

(416) 235-5842
FAX: (416) 235-5744

Laboratory Services Branch
Quality Management Unit
125 Resources Road
Rexdale, Ontario
M9W 5L1
(416) 235-5842
FAX (416) 235-5744

June 5, 1990

TO: PARTICIPANTS OF INTERLABORATORY STUDY 90-5

Please find enclosed four 250 mL glass bottles. The samples are labelled as follows:

VOC1 VOC2 VOC3 VOC4

If you are missing any of the above items, please contact us at the above phone number immediately.

As stated in the notification distributed May 10, 1990, samples should be analyzed using the principles and protocols outlined in the MISA General Regulation (Ontario Reg. 695/88, as amended to Ont. Reg. 533/89). Store all samples in a refrigerator at 4 degrees Celcius until ready for analysis. Time limit for storage is 7 days.

To ensure timely release of a summary report, results are to be submitted by July 13, 1990. Report forms to be used are included with the samples. Please identify all sample results with your lab identification number and the sample numbers described above. Please contact us if there are any problems or questions re the interlaboratory study. Thank you for your participation.

Your lab identification number is:

Sincerely,

Sylvia Cussion
Lab Quality Audit Scientist
(416) 235-5842

INTERLABORATORY STUDY 90-5

REPORT FORM

REPORT DUE DATE: JULY 13, 1990

LABORATORY IDENTIFICATION CODE:

PARAMETER	VOC1	VOC2	VOC3	VOC4
Benzene				
Toluene				
o-Xylene				
m- and p-Xylenes				
Acrylonitrile				

SAMPLE PREPARATION PRINCIPLES:

INSTRUMENTAL MEASUREMENT METHOD PRINCIPLES:

GAS CHROMATOGRAPHIC COLUMN:

Laboratory Services Branch
Quality Management Unit
125 Resources Road
Rexdale, Ontario
M9W 5L1
(416) 235-5842
FAX (416) 235-5744

June 5, 1990

TO: PARTICIPANTS OF INTERLABORATORY STUDY 90-5

Please find enclosed eight 250 mL glass bottles. The samples are labelled as follows:

VOC1 VOC2 VOC3 VOC4

You should have received two of each sample. If you are missing any of the above items, please contact us at the above phone number immediately.

As stated in the notification distributed May 10, 1990, samples should be analyzed using the principles and protocols outlined in the MISA General Regulation (Ontario Reg. 695/88, as amended to Ont. Reg. 533/89). Store all samples in a refrigerator at 4 degrees Celcius until ready for analysis. Time limit for storage is 7 days.

To ensure timely release of a summary report, results are to be submitted by July 13, 1990. Report forms to be used are included with the samples. Please identify all sample results with your lab identification number and the sample numbers described above. Please contact us if there are any problems or questions re the interlaboratory study. Thank you for your participation.

Your lab identification number is:

Sincerely,

Sylvia Cussion
Lab Quality Audit Scientist
(416) 235-5842

Laboratory Services Branch
Quality Management Unit
125 Resources Road
Rexdale, Ontario
M9W 5L1
(416) 235-5842
FAX (416) 235-5744

August 30, 1990

TO: PARTICIPANTS OF INTERLABORATORY STUDY 90-5

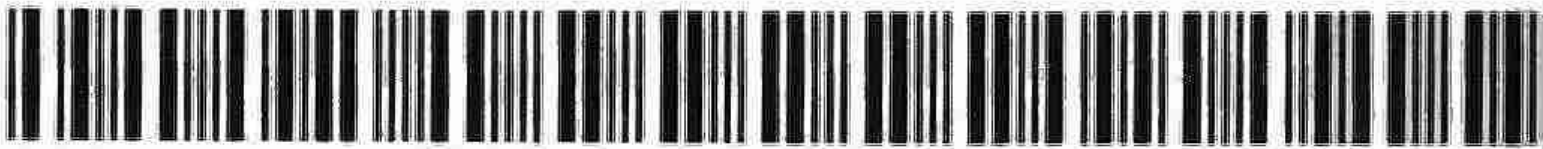
Thank you for your participation in Interlaboratory Study 90-5 for the analysis of Benzene, Toluene, Xylenes, and Acrylonitrile in reagent water. Attached is the table of results from all of the participants, identified by laboratory code. Please review your data for transcription errors and report any changes to me by September 14, 1990. After this date, all results will be considered final and will be used to prepare the final report. All participants will receive a copy of the final report as soon as it becomes available.

Please contact me if you have any questions.

Your laboratory identification code is:

Sincerely,

Sylvia Cussion
Lab Quality Audit Scientist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